NASA-CR-54007

40p.

N64-19944
CODE-1

MATERIALS FOR POTASSIUM LUBRICATED JOURNAL BEARINGS

Quarterly Progress Report No. 2
For Quarter Ending October 22, 1963

EDITED BY R. G. FRANK

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-2534

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI 15, OHIO

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MATERIALS FOR POTASSIUM LUBRICATED JOURNAL BEARINGS

QUARTERLY PROGRESS REPORT 2

Covering the Period
July 22, 1963 to October 22, 1963

edited by

R. G. Frank
Program Manager

approved by

J. W. Semmel, Jr.
Manager, Materials and Processes

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2534

Technical Management
NASA - Lewis Research Center
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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
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FOREWORD

The work described herein is being performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2534. Its purpose, as outlined in the contract, is to evaluate materials suitable for potassium lubricated journal bearing and shaft combinations for use in space system turbogenerators and, ultimately, to recommend those materials most appropriate for such employment.

R. G. Frank, Manager, Physical Metallurgy, Materials and Processes, is administering the program for the General Electric Company. L. B. Engel, Jr., D. N. Miketta, T. F. Lyon, W. H. Hendrixson and E. M. Bamberger are directing the program investigations. The design for the friction and wear tester is being executed by H. H. Ernst.

R. L. Davies of the National Aeronautics and Space Administration is the technical manager for this study.

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I. INTRODUCTION

The program reviewed in this second quarterly report, covering activities from July 22, 1963 to October 22, 1963, is performed under the sponsorship of the National Aeronautics and Space Administration. Its purpose is to evaluate materials suitable for potassium lubricated journal bearing and shaft applications in space system turbogenerators operating over a 400°F to 1600°F temperature range. The critical role of bearings in such systems demands the maximum reliability attainable within today's state-of-the-art. Achieving this reliability requires an interdisciplinary approach employing the best mechanical designs of journal bearings combined with the selection of the optimum materials to serve as the structural members. Satisfying this latter requirement constitutes the aim of this program.

A number of investigators have conducted studies in this field and their contributions have advanced the state-of-the-art considerably.[1] Although their work is significant, there are no common criteria for a comparison of the existing data. Therefore, establishing a unified approach to the development and evaluation of materials for potassium lubricated bearing application is deemed essential. The program involves a comprehensive investigation of material properties adjudged requisite to reliable journal bearing operation in the proposed environment. This includes: 1) corrosion testing of individual materials and potential bearing couples in potassium liquid and vapor, 2) determination of hot hardness, hot compressive strength, modulus of elasticity, thermal expansion and dimensional stability characteristics, 3) wetting tests by potassium, and 4) friction and wear measurements of selected bearing couples in high vacuum and in liquid potassium.

Applying a compilation of existing data on available materials, candidate materials will be selected in cooperation with the cognizant NASA technical manager. The materials reviewed fall into four broad categories:

- Superalloys and refractory alloys with and without surface treatment.

- Commercial metal bonded carbides.

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[1]"Materials for Potassium Lubricated Journal Bearings," Qtr. Rept. 1, Ctr. NAS 5-681 (July 22, 1963), SPPS, MSD, General Electric Company. v. Section VIII. Bibliography.

- Refractory compounds such as stable oxides, carbides, borides and nitrides.

- Cermets based on the refractory metals and stable carbides.

Each material will be procured from appropriate suppliers to mutually acceptable specifications and subsequently subjected to chemical, physical and metallurgical analyses to document its characteristics before utilization in the program. After the documentation of processes and properties, the candidate materials will undergo corrosion, dimensional stability, thermal expansion, compression and hot hardness testing. Considering the bearing material requirements and the preliminary information obtained on materials subjected to both potassium and non-potassium testing, a number of materials combinations will be selected in cooperation with and subject to the approval of the NASA technical manager. Potassium corrosion and wetting tests and friction and wear measurements in high vacuum and liquid potassium will then proceed wtih these combinations.

The ultimate product of this program will be a recommendation, substantiated with complete documentation, of the material or materials which have the greatest potential for use in alkali metal journal bearings in high speed, high temperature, rotating machinery for space applications. Hopefully, the results will indicate the future course of alloy or material development specifically designed for alkali metal lubricated journal bearing and shaft combinations.

II. SUMMARY

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During the second quarter of the contract, the topics abstracted below were covered. The results are interpretatively presented in this report.

NASA approved fourteen materials for inclusion in the program. These include one nonrefractory alloy, two refractory metals and alloys, three nonrefractory metal bonded carbides, four refractory metal bonded carbides and four pure compounds.

The literature search and data compilation of properties of the candidate materials was continued. When completed, the data will be published in a topical report.

In an effort to establish mutually agreeable chemistry and property limitations and quality assurance provisions to assist in preparing procurement specifications, discussions were held with nine vendors.

Preliminary test plans covering seven test programs were submitted to NASA for approval. These include: Potassium Purification and Analyses, Corrosion, Dimensional Stability, Hot Hardness, Compression, Thermal Expansion and Friction and Wear in Vacuum.

The proposed procedure for the purification and analyses of potassium was approved by NASA. Component parts of the purification train have been ordered.

The design for the isothermal capsule corrosion facility was completed and detail drawings are being prepared.

The design for the dimensional stability test program was initiated.

The fabrication of the vacuum chamber for the compression testing facility was completed.

The design for the high vacuum friction and wear tester was completed and detail drawings are in process. The high vacuum chamber and pumping system was checked out at 6 x 10^{-10} torr cold, dry and empty.

Author

The design for the liquid potassium version of the friction and wear tester was initiated.

Two progress meetings, August 15, 1963 and September 2 , 1963, were held with the NASA technical manager during the reporting period.

III. MATERIALS SELECTION

The National Aeronautics and Space Administration endorsed fourteen candidate materials to be included in this program. The list includes the following materials selected from an extensive compilation on thirty-five materials submitted to NASA under separate cover on July 24, 1963 and recorded in the first quarterly progress report.

1. Star J
2. TZM Mo
3. Tungsten
4. K601 Carbide
5. 999 Carbide
6. 907 Carbide
7. TiC
8. Al_2O_3
9. ZrO_2
10. TiB_2
11. TiC + 5% W
12. TiC + 10% Mo
13. TiC + 10% Cb
14. Proprietary

A constant effort is being devoted to compiling and referencing existing data on properties of potential bearing materials. Additional data were obtained during this interim for these materials:

- TiN
- Ces
- Ta_2Be_{17}
- Al_2O_3
- TiB_2
- TiC
- ZrC
- Y_2O_3
- ZrO_2
- $CbSi_2$

The characteristics being surveyed are hardness; compressive strength; elastic modulus; thermodynamic properties, i.e., heat of formation, entropy, free energy, specific heat; density; melting point; thermal expansion; thermal conductivity; crystal structure; vapor pressures, and evaporation rates. When sufficient data have been compiled, they will be edited and incorporated in a topical report. To be recognized is the fact that the reliability of much of the data is dubious because of poor or unreported experimental techniques and/or poor quality materials which were used. Frequently, the quality of the original material was never investigated and the data were reported without record of this fact.

IV. MATERIALS PROCUREMENT

Columbium-1% Zirconium Alloy

Four hundred feet of 0.062-inch diameter Cb-1Zr wire was received from the Kawecki Chemical Company. This material will be used to fabricate specimen holders for properly positioning the corrosion samples in the liquid and vapor regions of the Cb-1Zr alloy capsules. The wire was purchased to the chemical, product quality and final heat treatment requirements of specification SPPS-1B, "Bar, Rod, Sheet, Plate, and Strip: Columbium-1% Zirconium Alloy," Space Power and Propulsion Section, General Electric Company.

The Kawecki Chemical Company certified that the material was produced from heat number 7B502-3 with the ingot analysis given in Table I. The data derived from the vendor's check analysis of the final product for interstitial elements are also presented in Table I. Note that the hydrogen content of the final product shows a considerable reduction from the level reported for the original ingot. The final product heat treatment of one hour at 2200°F in a vacuum of 10^{-4} torr or greater was probably instrumental in lowering the hydrogen content to the specified product analysis limits of 0.0010%. A check analysis of a sample taken approximately at the midpoint of the 400-foot strand was performed by the General Electric Company and verified the vendor's analysis as shown in Table I.

The Cb-1Zr alloy sheet, 0.080-inch thick x 4-inch wide x 15-inch long, previously rejected because of ultrasonic indications, was replaced by the Kawecki Chemical Company. The material, produced from heat number 7B506, had the following chemical analysis:

Chemical Analysis of Cb-1Zr Alloy Sheet

Element	Product Analysis, wt %
Zirconium	1.20
Carbon	0.0080
Oxygen	0.0300
Nitrogen	0.0012
Hydrogen	0.0007

Automation Industries, Columbus, Ohio, performed an ultrasonic inspection of this replacement stock, which conformed to the requirements of specification SPPS-1B. Purchaser check analysis of the interstitial elements contained in both this replacement sheet and in the original sheet are in process.

TABLE I: CHEMICAL ANALYSES OF Cb-1Zr WIRE
(Heat Number 7B502-3)

Element	Ingot Analysis, wt %	Final Product Analysis, wt % Kawecki 169994	GE 460
Zirconium	1.03	---	---
Carbon	0.0055	0.0042	0.005/0.007
Nitrogen	0.0040	0.0042	0.0030
Oxygen	0.0200	0.0172	0.0158
Hydrogen	0.0040	0.0007	0.007
Tantalum	<0.0800	---	---
Titanium	0.0010	---	---
Silicon	0.0020	---	---
Iron	0.0030	---	---

Production of the 1.0-inch diameter x 0.080-inch thick wall tubing for fabrication of the corrosion capsules has been delayed by the vendor's subcontractors. The delivery date is now November 1, 1963, a three month delay from the original promise date. The lack of acceptable vacuum heat treating facilities for intermediate and final annealing of refractory alloy mill products in lengths exceeding several feet has proved the major obstacle in the production process. When the tubing is received and processed through the specified quality assurance tests, all the Cb-1Zr raw materials required for fabrication of the corrosion capsules will be on hand.

Bearing Materials

Producers of the materials which were approved by NASA were apprised of the selection. Subsequently, visits to obtain the information and data prerequisite to detailing procurement specification were arranged. The problem of reproducibility motivated a decision to concentrate on those vendors, approximately 60% of the list, whose products necessitated powder metallurgy techniques in the production processes. This class of materials required that explicit vendor-purchaser agreements be established to ensure, as nearly as possible, the eventual procurement of sound, representative and reproducible material. Producers of the less known wrought materials, i.e., pure cobalt and Vasco-Hypercut, were also visited. Table II is a listing of the vendors who were contacted and the materials which were discussed.

The discussions with the vendors concentrated on four fundamentals:

1) A review of the project goals, emphasizing particularly the requisite to achieve the highest quality and reproducibility in their respective materials

2) The vendors experience with those production processes which, in their opinion, most likely realize the quality of product compulsory for the test program

3) The materials properties

4) The quality control measures to be specified in forthcoming purchase orders.

The production method selected for the majority of the powder metallurgy materials entailed cold pressing master blocks of the material, sectioning the blocks into specimen blanks and, finally, sintering and grinding the blanks into final configurations.

Efforts to establish quality control provisions in the specifications engendered considerable opposition from many of the vendors. Most of

TABLE II: VENDORS CONTACTED FOR THE PREPARATION OF PROCUREMENT SPECIFICATIONS

Vendor	Candidate Material
GE, Metallurgical Products Dept. Detroit, Michigan	Carboloy 779, 907, 999
Kennametal, Inc. Latrobe, Pennsylvania	K601, K150A, TiC and Refractory Metals Bonded TiC
Carborundum Co. Niagara Falls, New York	ZrC, TiC, TiB_2
Firth-Sterling, Inc. Pittsburgh, Pennsylvania	ZrC, TiC, TiB_2 and Refractory Metals Bonded TiC
GE, Lamp Glass Dept. Cleveland, Ohio	Lucalox
Zirconium Corp. Cleveland, Ohio	Al_2O_3, ZrO_2, Y_2O_3
Vanadium Alloy Steel Co. Latrobe, Pennsylvania	Vasco-Hypercut
Stellite Div., Union Carbide Corp. Kokomo, Indiana	Star J and Cobalt
Norton Co. Worchester, Massachusetts	Al_2O_3, TiN, TiB_2, TiC, ZrC

the detailed processing and control procedures are veiled by vendor proprietary policies and material properties pertinent to a specification were provided quite reluctantly. In no case were any strength properties, transverse rupture, compression, etc., proferred as a control factor in procurement. Generally, the only properties to which the powder metallurgy material vendors consistently agreed for specification purposes were density, hardness and porosity, and, in cases of the lesser used materials, even these properties were unavailable.

Recognizing this limited information, a general procurement specification was prepared so that all the powder metallurgy materials could be ordered to a uniform specification by inserting the composition and available properties of a particular material. The specification was heavily weighted in the areas of raw material and process control documentation and in final product uniformity relative to chemistry, density, hardness and porosity as determined by destructive (test coupons) and nondestructive (test specimens) techniques. The specification permits latitude in the selection of the basic consolidation process, contingent upon the experience of the respective vendor, to produce materials with properties which the vendors consider representative and reproducible. To delineate limits for density, hardness and porosity for those materials lacking data, the specification requires several runs utilizing the more promising procedures to produce a reproducible maximum density. With this approach, additional samples would be fabricated to establish an average hardness and porosity for the specified properties for that material.

Because of the restrictiveness, documentation and quality assurance provision prescribed in the specification, substantial opposition may be encountered. However, to assure procurement of sound, representative and reproducible materials for the program, the principal contradictions, in all probability, can be mutually resolved.

A specification was prepared for the cast material Star J which, with few exceptions, approximates the general specification that was prepared for the powder metallurgy material. Specific property limits required in the specification were incorporated after consultation with representatives of the Stellite Division of Union Carbide Corporation.

By augmenting existing specifications to provide for the necessary documentation and quality assurance section, specifications for the wrought materials will be adopted.

V. TEST PROGRAM

Preliminary test plans for the following investigations were submitted to the NASA technical manager for review and approval July 30, 1963:

1) Potassium Purification and Analysis

2) Corrosion

3) Dimensional Stability

4) Hot Hardness

5) Compression

6) Thermal Expansion

7) Friction and Wear in Vacuum

Currently, the proposed procedures for the purification and analysis of the potassium, which is essential for the test programs, have been approved and construction of the purification system has been initiated. A description of the purification and analytical procedure is given in Section VI.

Test plans for the remaining two investigations, wetting and friction and wear in liquid potassium, will be detailed when the testing procedures have been defined.

VI. TEST FACILITIES

Potassium Purification and Analysis

The objective of this task is to supply potassium of the highest purity for three test programs: 1) capsule corrosion tests to determine the corrosion resistance of the candidate materials in potassium liquid and vapor 2) an investigation of frictional and wear properties in potassium liquid and, 3) since the lubricating properties of liquids are quite dependent on the surface properties of the liquid-substrate interface, a concurrent program will investigate the wetting characteristics of liquid potassium. The use of high purity potassium is warranted for several reasons. For example, the interstitial elements such as carbon, oxygen, nitrogen and hydrogen in the potassium are gettered by many materials, thereby changing their physical and mechanical properties. Also, the presence of oxygen in alkali metals at elevated temperatures intensifies the mass transfer of carbon[2] and some metallic elements.[3] Further, by analogy with other liquid systems, it is reasonable to anticipate that interfacial properties will be affected by small amounts of impurities which preferentially concentrate at interfaces or surfaces because of the changes in cohesive forces occurring at these locations. The presence of surface films on the contact surfaces has been affirmed, to markedly affect wettability and frictional forces.[4]

Test Equipment. The diagram in Figure 1 illustrates basic equipment and apparatus to be used for the potassium purification. The components include:

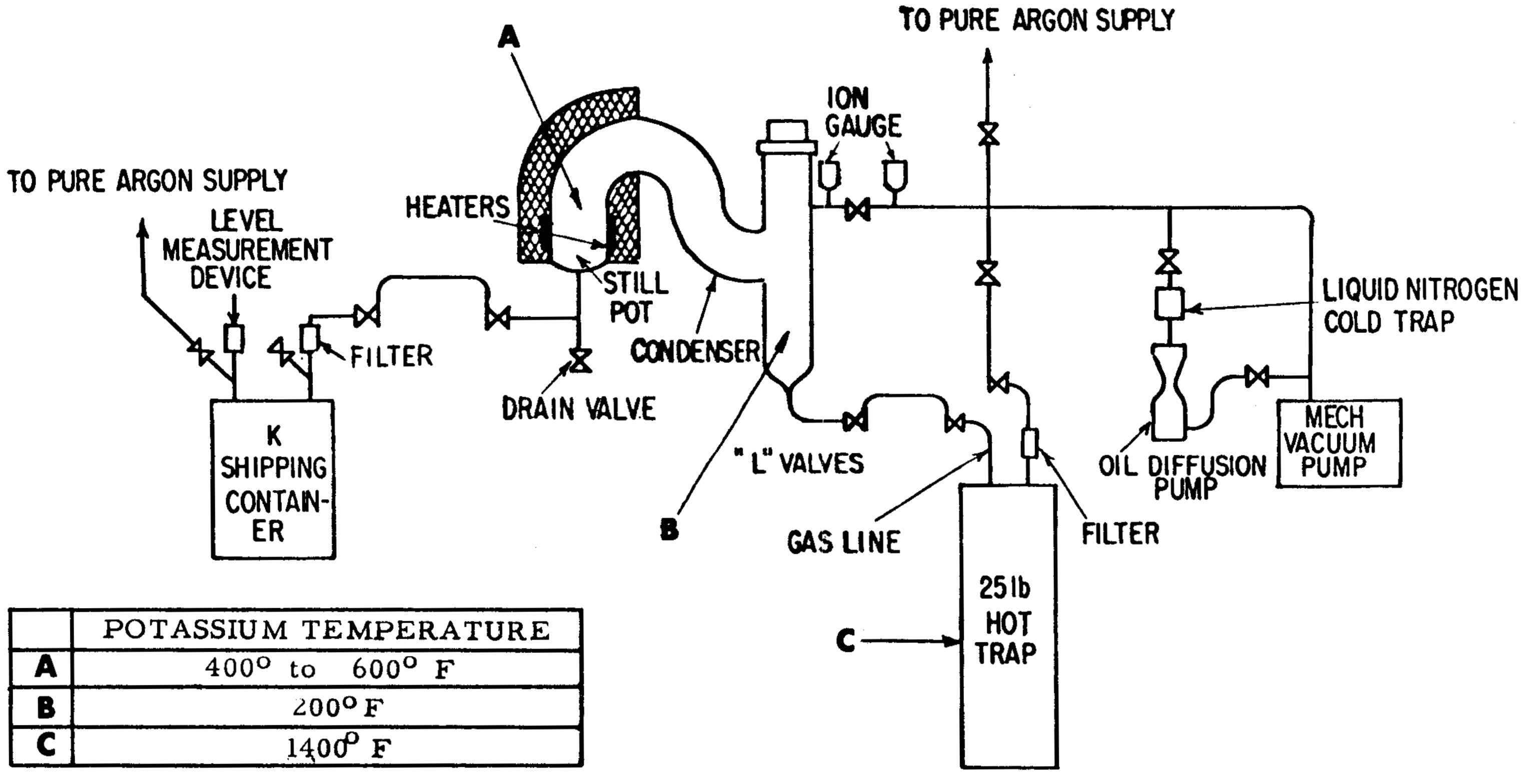
1) The potassium supply container in which the potassium will be outgassed and from which the material will be transferred to the still.

2) The vacuum distillation rig in which the potassium will be separated from its impurities at 450°F to 650°F and a vacuum of 10^{-5} torr in the receiver.

3) The titanium-lined, zirconium-gettered hot trap which will receive and getter the distillate at 1400°F.

4) The vacuum system which will be used for outgassing and distilling the potassium.

Auxiliary equipment and apparatus will include an argon purification system, temperature measurement equipment, vacuum gauge readout instrumentation and leak detection apparatus. The basic and auxiliary



	POTASSIUM TEMPERATURE
A	400° to 600° F
B	200° F
C	1400° F

Figure 1. Diagram of Potassium Purification Train

systems are described in the succeeding paragraphs.

Potassium Supply System. The shipping container in which the potassium will be outgassed and from which the potassium will be transferred to the still is shown in Figure 2. The shipping container will consist of a cylindrical can equipped with gas and potassium transfer piping. The potassium dip-leg outlet section will include a 5-micron, sintered, stainless steel filter and a stainless steel, bellows sealed, L-type valve that can be thoroughly cleaned down to the valve seat. The container gas line will be connected to a pure argon supply. The vertical gas port will have an insulated, sliding, quad-ring seal through which the potassium height can be measured with a 1/4-inch OD thermowell. The measurement will involve the completion of an electrical circuit when the thermowell touches the potassium; at this point the height of the thermowell extending above the gas port will be measured. The difference between the initial height and subsequent height measurements will represent the change in potassium level. The shipping container and transfer lines will be heated with heating tape.

Vacuum Distillation Rig. The distillation apparatus is shown in Figure 3. The design of the still is based on the performance of a distillation unit that has been used in the study of potassium purification in this laboratory.[5] Except as noted in Figure 3, all parts will be constructed from Type 304 SS.

The still pot will be constructed from a 5-inch diameter pipe cap and a 3-inch section of 5-inch diameter Schedule 10 pipe. The bottom of the pipe cap will be drilled to accept a 1/2-inch diameter Schedule 40 tee. A drain valve and a filling and sampling valve will be connected to the bottom tee with 1.2-inch diameter Schedule 40 pipe. The drain valve and the filling and sampling valve will be a stainless steel, bellows sealed type. Heating the still pot will be accomplished from the sides with three cylindrical band heaters, each rated at 750 watts.

The vapor path between the boiler and condenser will be formed by a 5-inch diameter Schedule 10, short radius, 90° elbow. The up-pass elbow will contain a thermowell which extends vertically down the center of the still pot into the tee at the bottom of the pot. The entire boiler and up-pass section will be insulated with 2 to 3 inches of aluminum-encased, fibrous alumina-silica. The condenser section will consist of two 5-inch diameter Schedule 10, long radius, 90° elbows shown in Figure 3. This section will be cooled by natural convection and radiation to the atmosphere.

As shown in Figure 3, the distillate receiver will consist of a 4-inch diameter Schedule 10 pipe connected to the condenser through an adapter plate. The distillate will flow into the receiver through

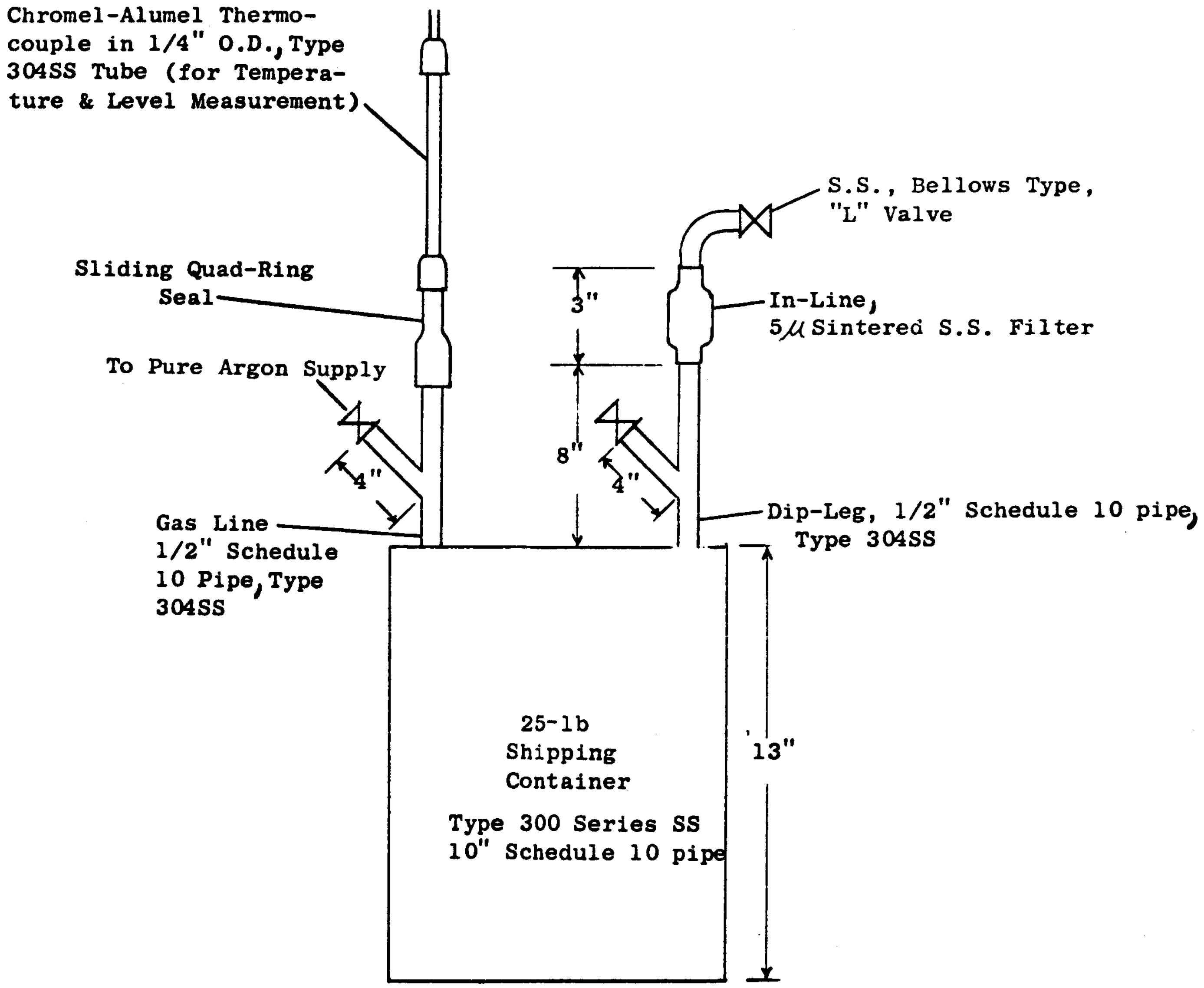


Figure 2. Potassium Supply System

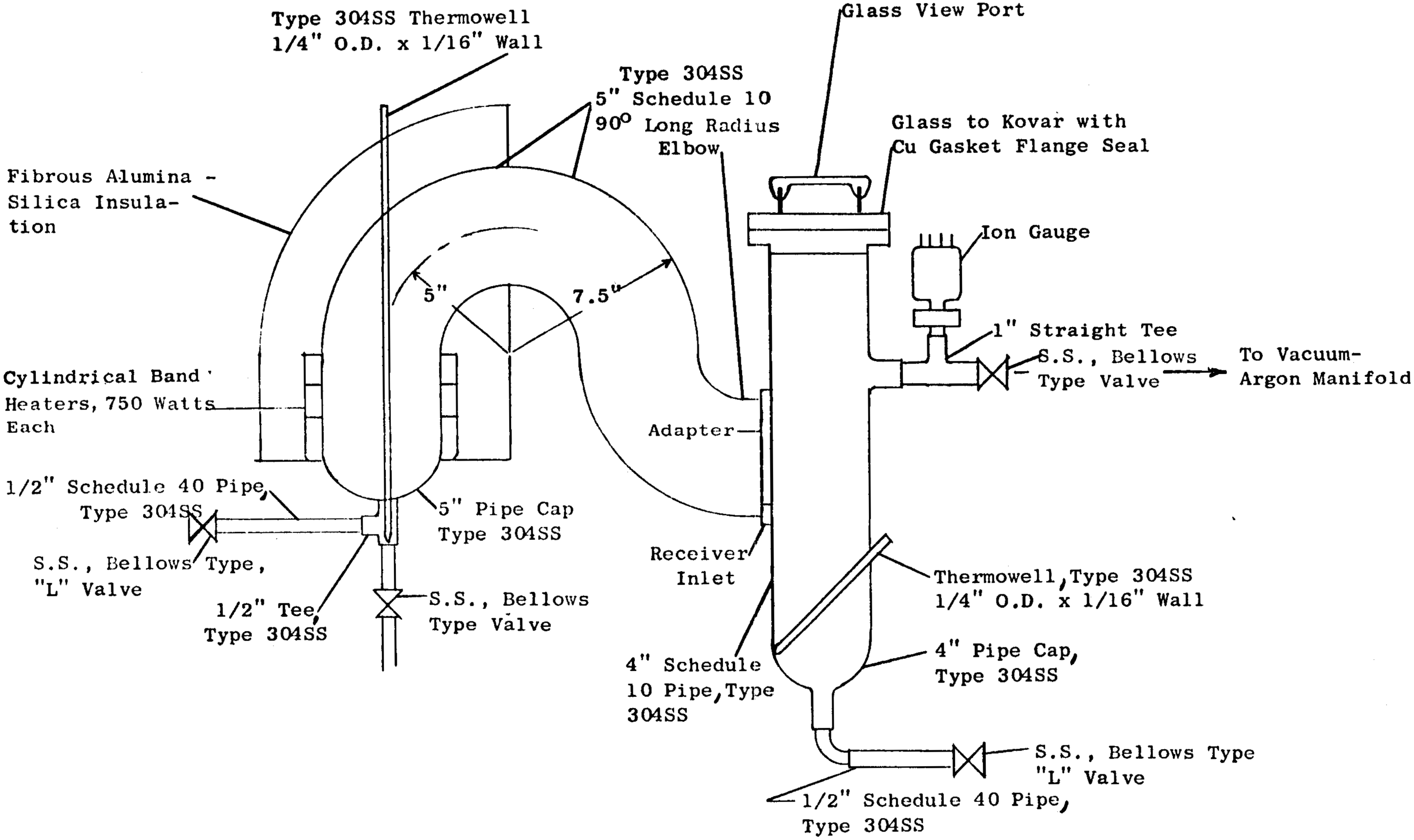


Figure 3. Distillation Apparatus for Purifying Potassium

a 1-inch diameter hole in the adapter plate and receiver wall. A glass viewport, a glass-to-Kovar seal assembly, will be attached to the top of the receiver with a copper-gasketed flange. The receiver section below the condenser will contain a thermowell for temperature and level measurement. The bottom of the receiver will be connected to a stainless, bellows sealed L valve by 1/2-inch diameter Schedule 40 pipe. The receiver vacuum port will be located approximately 6 inches above the distillate inlet and will contain vacuum measurement gauges and a stainless steel, bellows sealed valve.

Hot Trap. A twenty-five-pound capacity hot trap, shown in Figure 4 and essentially identical to the type currently being used in this laboratory for potassium purification work, will receive the distillate.[5] The body will be a 30-inch length of 8-inch diameter Schedule 10, Type 316 SS pipe equipped with 1/4-inch thick end plates. A dip leg containing a filter and a stainless steel, bellows sealed L valve extends to within one inch of the bottom of the hot trap. The gas line is also equipped with a stainless steel, bellows sealed L valve because the distillate will enter the hot trap through this valve and necessitate cleaning. The titanium liner will be fabricated from 0.040-inch thick titanium sheet of grade Ti55A or purer. Alternate straight and corrugated strips of zirconium will form the getter bundle, which will be attached to the dip leg by spot welding. The hot trap will be heated with electric heating units with a 9 kw total power rating. Insulating will be accomplished with fibrous alumina-silica.

Vacuum System. Figure 5 shows the vacuum system to be used for outgassing and distillation. The system will include a 140ℓ/sec high capacity roughing pump and a 2-inch, 85ℓ/sec fractionating oil diffusion pump. The inlet to the diffusion pump will be baffled by a cold trap, cooled with liquid nitrogen, to minimize backstreaming of oil vapor. All valves upstream from the vacuum system will be the stainless steel, welded bellows type.

Fabrication and Assembly. With those exceptions indicated in the illustrations, all joints in the distillation, hot trap and vacuum systems will be fusion welded using tungsten-inert-gas techniques. Before welding, all parts will be degreased and then pickled in 20% aqueous hydrochloric acid and, whenever feasible, all welds will be pickled in the acid solution to remove surface oxides. Potassium transfer lines between the shipping container and still pot and between the still receiver and hot trap will consist of 1/2-inch OD, Type 321 SS tubing and Type 316 SS swagelok connectors. If properly installed, experience has shown that such connectors do not leak at the temperature required for transferring the potassium.

Argon Purification. A special, high purity grade argon cover gas will be used. The gas will be purified further in a system which

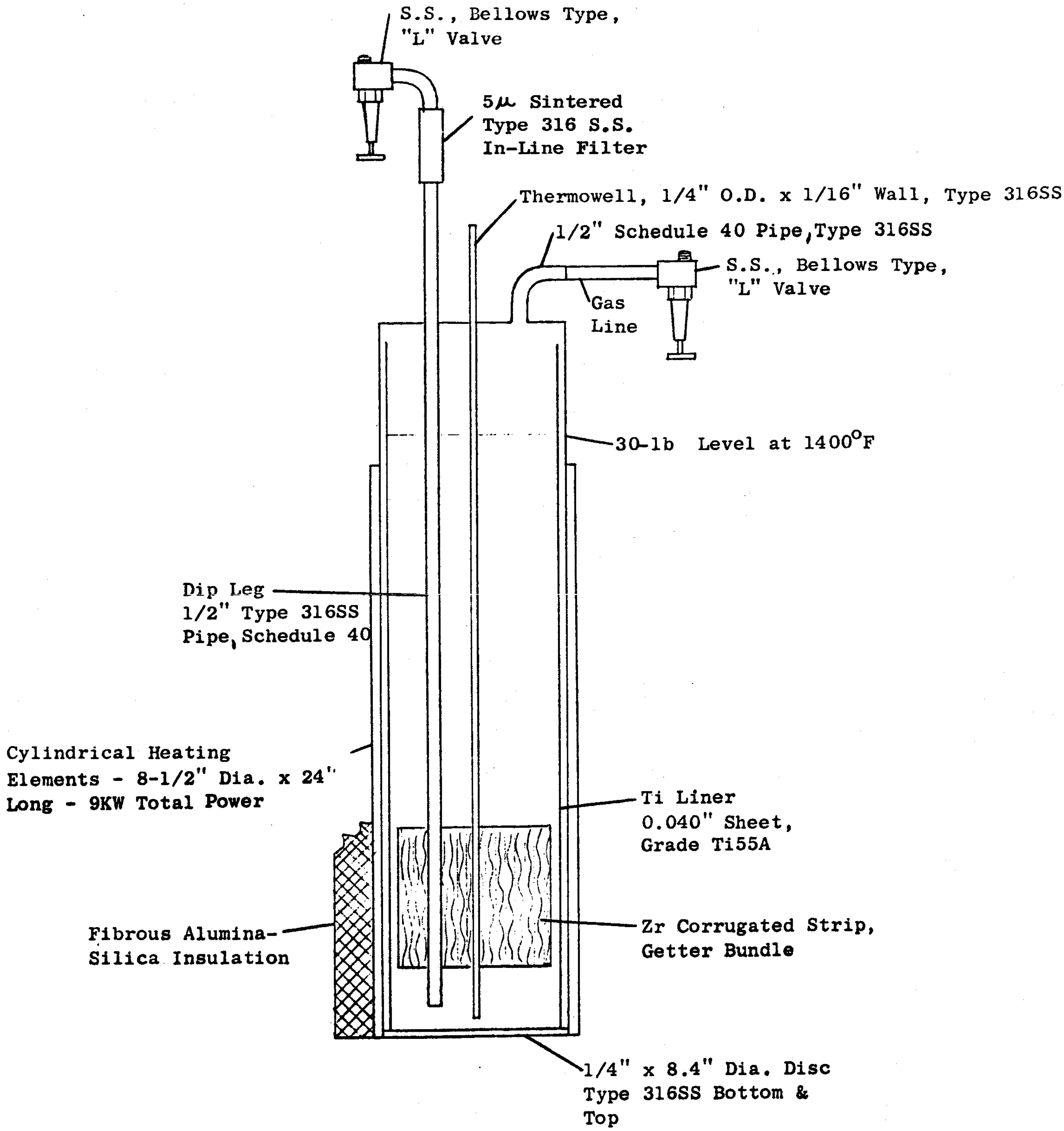


Figure 4. Twenty-Five-Pound Capacity Hot Trap for Purifying Potassium

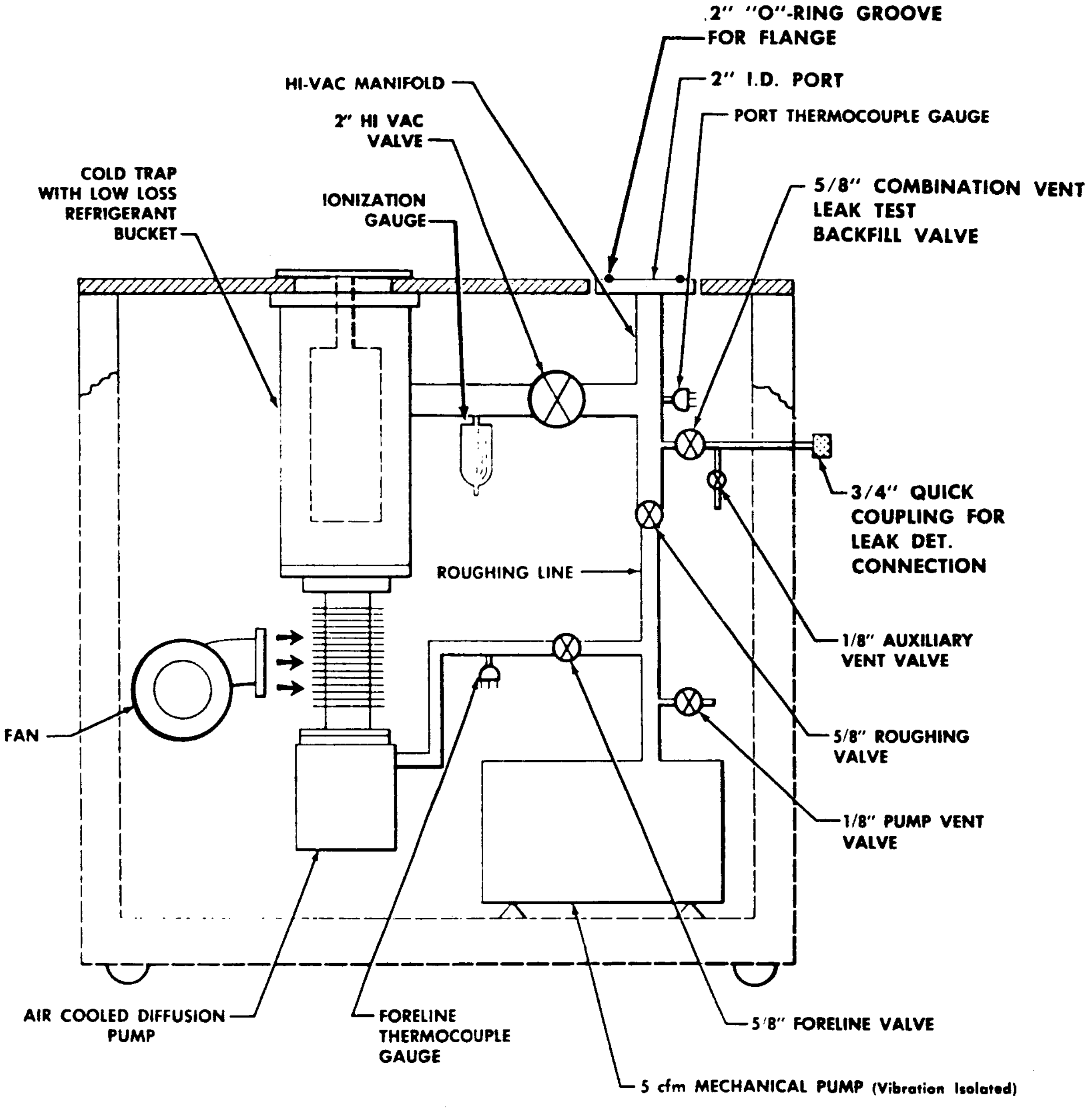


Figure 5. Vacuum System for Outgassing Potassium Purification Train and Distillation of Potassium

is identical to one currently operating at this laboratory. The procedure entails passing the gas through a stainless steel, 9 Å molecular sieve and, subsequently, over titanium turnings heated to 1500°F. After the argon is passed through this system, it should have an oxygen content of <0.6 ppm by volume measured by the Brady apparatus and a water content of <1 ppm by volume measured by a dew point indicator.

Temperature Measurement. Chromel-alumel, MgO insulated, stainless steel sheathed thermocouples will be used for all potassium temperature measurements. Calibration of the thermocouples will be accomplished by comparing them with a platinum resistance thermometer in an oil bath up to 650°F and with a standardized Pt vs. Pt + 10% Rh thermocouple above 650°F. Temperatures will be read out on a multipoint potentiometric recorder.

Leak Checking. After fabrication, the distillation and hot trapping apparatus will be helium leak checked with a mass spectrometer leak detector. The maximum sensitivity of this instrument is approximately 10^{-8} atm-cc/sec.

Pressure Measurement. Thermocouple vacuum gauges will measure pressures down to 10^{-3} torr. Pressures from 10^{-3} torr to 10^{-6} torr will be measured by Bayard-Alpert ionization gauges.

Test Procedures. Potassium with a guaranteed analysis of less than 50 ppm oxygen and less than 50 ppm sodium can be purchased. This grade potassium will be used in this program.

After the potassium is delivered, the following purification procedures will be applied:

1) The potassium will be heated to 450°F in the shipping container and degassed by evacuating with the 140 ℓ/sec mechanical pump through a dry ice-acetone cooled cold trap until the pressure becomes constant.

2) The potassium will be backfilled with purified argon, cooled slowly to 200°F and filtered into the still pot through a 5 10 micron pore size, stainless steel, micrometallic filter. Previously, this still will have been helium leak checked, outgassed, and again leak checked at a pressure below 10^{-5} torr. The pressure will be measured on the still size of the cold trap.

3) The still pot, which will not hold the total amount of potassium distilled in one run, will be continuously refilled from the bottom during the

distillation cycle. Surface evaporation will be maintained between 450° and 600°F. Depending on the receiver temperature, the pressure in the still will vary from approximately 1 torr in the pot to approximately 10^{-5} torr in the receiver.

4) The potassium in the receiver will be continuously emptied into a titanium lined, zirconium gettered hot trap, until the hot trap is full (25-30 lbs).

5) The potassium will be gettered for more than 50 hours at 1400°-1500°F before being transferred to any test rig or capsule.

6) The potassium will be analyzed before and after purification and also when it is transferred.

Since, with this purification system, the final purity of the potassium produced depends primarily upon the purification procedure rather than the initial potassium quality, the potassium which has been used in the friction tester will be reclaimed. The purification equipment described in the preceding discussion has been designed in detail and components have been ordered.

The as-received potassium will be analyzed for oxygen and sodium. After purification, a set of analyses will be obtained for oxygen, nitrogen, metallics and, possibly, carbon. A carbon analysis will be performed only if a suitable method has been developed. Additionally, analyses for oxygen will be performed on samples obtained during the transfer of all potassium to either the capsule or friction test equipment and analyses for metallics will be performed on the potassium obtained after test from the capsules tested at the maximum test temperature. Further, if, because of the test schedule, the purified potassium remains in the hot trap longer than two weeks between uses, it will be re-analyzed for oxygen before transfer to capsule or friction test equipment.

Currently, the interstitial elements, oxygen and nitrogen in the 0 to 50 ppm range, can be analyzed with precision (± 5 ppm) on properly obtained specimens; a method for the determination of total carbon is expected to be developed completely in the near future. Overall precision for the latter has not been established yet, but present evidence indicates a precision similar to that for oxygen and nitrogen.

Oxygen is determined by the amalgamation technique and, since the true nature of the residue by amalgamation is unknown, the accuracy of the "oxygen" values obtained is not known. However, a low "oxygen" value indicates high purity, since it correlates with the purification procedures applied. Nitrogen is analyzed by a modified micro-Kjeldahl technique. The analytical method for carbon is an adaptation of the combustion method described by S. Kallmann and R. Liu of Ledoux and Company, Incorporated. The metallic impurities in potassium will be analyzed by spectrographic techniques. Present capabilities for the analysis of the metallic impurities in potassium, such as potassium chloride, are in the 0 to 25 ppm range. During the first quarter of 1964, however, this range will be extended. Table III presents the detection limits. Flame photometric techniques will be used to analyze potassium for sodium

Corrosion

The design of the test setup for conducting the isothermal capsule corrosion tests is completed essentially and detailed drawings are being prepared for submission to the NASA technical manager during the next quarter. On September 27, 1963, a drawing of the conceptual design was forwarded to NASA for review. Details of the facility will be reported after NASA approval.

Dimensional Stability

The design of the test facility to be employed in evaluating the dimensional stability of the candidate materials is currently underway. The basic design approximates that tentatively planned for the isothermal corrosion test program. Detailed drawings should be prepared and submitted to the NASA technical manager within the next reporting period.

Hot Hardness

To measure the hot hardness of the candidate materials, facilities located at the General Electric Company's Research Laboratory will be used. The instrument, designed and fabricated by Research Laboratory personnel, has provided valuable data on a variety of materials ranging from glasses, ceramics and intermetallic compounds to pure metals. However, since the consistent operating vacuum is approximately 1.0×10^{-4} torr, less than the 10^{-6} torr level desirable for the program, a meeting was held at the Research Laboratory to discuss the feasibility of improving the vacuum capabilities of the instrument. The general agreement was that a complete major overhaul of the facility will be executed in an attempt to achieve the required vacuum. When this has been accomplished, a trial run will be conducted with a Cb-1Zr alloy specimen to determine the acceptability of the facility.

Compression

In August, an approved vendor was selected for the vacuum chamber which is being purchased for the program by the General Electric

TABLE III: SENSITIVITY OF SPECTROGRAPHIC ANALYSIS FOR METALLIC IMPURITIES IN POTASSIUM
(As Chloride)

Element	Min. Amt. Detectable
Mg	<1
Mn	<1
Cr	<1
Sn	1
Si	<1
Ti	1
Al	<1
V	1
Cu	5
Ag	<1
Co	<1
Ni	1
Zr	5
Pb	5
Mo	<1
Ca	<1
Cb	1
B	5
Fe	1
Na	1
W	5
Ba	Undetected to 25 ppm
Be	<1
Sr	<1
Ta	5

Company to be used for the compression testing facility. Figure 3 in the first quarterly report is a schematic of the chamber. The chamber has been fabricated and will be delivered to General Electric in late October. Too, major accessory equipment required for the facility, including step-down transformers, a sealed linear-variable-differential transfer for strain measurement and a silicon controlling rectifier for temperature control, has been received. The design and selection of materials for the loading train components also progressed. Either K601 or Carboloy 883 were selected tentatively for the platen material. The loading heads within the hot zone of the furnace will be fabricated from TZM molybdenum alloy and the sections operating in the cooler areas will be made of Rene' 41 alloy.

Thermal Expansion

The test techniques required to operate the Chevenard dilatometer were reviewed, and the instrument's precision and reliability were established by examining accumulated data and reports from previous investigations. Several alternate methods were proposed for conducting the test under an inert atmosphere and the necessary modifications were evaluated for simplicity and reliability in producing the desired environment. These alterations were incorporated into the preliminary test plan submitted to NASA and, after NASA approval, will be reported.

Friction and Wear in Vacuum

The design of the high vacuum friction and wear tester has been completed, and preliminary engineering drawings were submitted to the NASA technical manager on September 27, 1963 for review. To assure the operational integrity of the tester, shaft stability and heat transfer calculations were included in the final design. With the existing tester configuration, the main shaft will operate approximately 20-25% below its first critical speed.

The materials that were selected for all structural components of the tester will conform to either current or modified AMS, ASTM, or GE specifications. Table IV is a list of selected materials. In addition, metallurgical and manufacturing process instructions have been prepared for those parts which, because of their critical nature, e.g., the shaft, or their complex fabrication, e.g., the vacuum chamber, require engineering coverage to assure high reliability.

TABLE IV: MATERIALS SELECTION FOR HIGH VACUUM FRICTION AND WEAR TESTER

Dwg. No.	Part Name	Material	Specification
SK-56131-250	Assembly Drawing		
SK-56131-251	Bearing Housing	Type 304 SS	AMS 5647A
SK-56131-252	Drive Shaft	Type 304 SS	AMS 5639A
SK-56131-253	Spacer-Stationary	Type 304 SS	AMS 5639A
SK-56131-254	Bearing Retainer	Type 304 SS	AMS 5513
SK-56131-255	Sleeve-Stationary	Al-Bronze	AMS 4630E
SK-56131-256	Grease Retainer	Type 304 SS	AMS 5513
SK-56131-257	Grease Retainer	Type 304 SS	AMS 5513
SK-56131-258	Spacer-Shaft	Type 304 SS	AMS 5639A
SK-56131-259	Spacer-Shaft	Type 304 SS	AMS 5639A
SK-56131-260	Sleeve-Rotating	Al-Bronze	AMS 4630E
SK-56131-261	Retainer	Type 304 SS	AMS 5639A
SK-56131-262	Shaft	M-252	GE B50T55D
SK-56131-263	Specimen Holder	TZM	SPPS 15
SK-56131-264	Specimen Holder	TZM	SPPS 15
SK-56131-265	Shield	M-252	GE B50T55D
SK-56131-266	Shoulder Bolt	M-252	GE B50T55D
SK-56131-267	Shim	Type 304L SS	AMS 5511A
SK-56131-268	Retainer	Type 304L SS	AMS 5647A
SK-56131-269	Cooling Coils	Type 304L SS	AMS 5560D
SK-56131-270	Bus Bar	Cu-OFHC	ASTM B187-55
SK-56131-271	Bus Bar Clamp	Cu-OFHC	ASTM B187-55
SK-56131-272	Heat Shield Cover	Ta	ASTM B364-61T
SK-56131-273	Heat Shield & Support	Type 304L SS	AMS 5647A
SK-56131-274	Heating Element	Ta	ASTM B364-61T
SK-56131-275	Vacuum Chamber	Type 304L SS	AMS 5647A
SK-56131-276	Diaphragm	Inconel X	GE B50R216
SK-56131-277	Magnet	Cast Alnico 5	GE B3C9E
SK-56131-278	Specimen Holder Assembly		
SK-56131-279	Washer	M-252	GE B50T55D
SK-56131-280	Nut	M-252	GE B50T55D
SK-56131-281	Gasket	Cu-OFHC	ASTM B187-55
SK-56131-282	Sleeve	Type 304 SS	AMS 5647A
SK-56131-283	Support	Type 304 SS	AMS 5639A
SK-56131-284	Nut	TZM	SPPS 15
SK-56131-285	Bearing Housing	Type 304 SS	AMS 5639A
SK-56131-286	Shim	Type 304 SS	AMS 5513
SK-56131-287	Gimbel	Type 304 SS	AMS 5639A
SK-56131-288	Sleeve	Al-Bronze	AMS 4630E
SK-56131-289	Bearing Housing	Type 304 SS	AMS 5639A
SK-56131-290	Shaft	Type 304 SS	AMS 5639A
SK-56131-291	Belt	Mylar	
SK-56131-292	Shim	Type 304 SS	AMS 5513
SK-56131-293	Shaft	Type 304 SS	AMS 5639A
SK-56131-294	Support	Type 304 SS	AMS 5639A
SK-56131-295	Test Specimen Holder Assembly		
SK-56131-296	Specimen Holder	Rene' 41	GE C50T62
SK-56131-297	Specimen Holder Extension	Type 304 SS	AMS 5647A
SK-56131-298	Flange & Bellows Assembly	Type 304 SS	AMS 5511A/AMS 5647A
SK-56131-299	Bellows	Type 304 SS	AMS 5560D
SK-56131-300	Shaft Wrench		
SK-56131-393	Magnet (Unmachined)	Cast Alnico 5	GE B3C9E

The materials for the major components of the tester were selected using the following criteria:

Material	Criteria
Type 304 SS	All structural parts not exposed to high vacuum environment.
Type 304L SS	All parts exposed to high vacuum and/or requiring metallurgical joining.
M-252	Parts requiring good high temperature strength and low thermal conductivity.
TZM	Parts requiring good high temperature strength and a low coefficient of thermal expansion. Since the parts made from TZM will contact the M-252 alloy, an additional consideration in the selection of TZM was minimizing diffusion bonding.
Inconel X	Parts requiring high tensile strength and high fatigue strength and low magnetic permeability.
Rene' 41	Parts requiring excellent high temperature strength.
Au plated M50 Ag plated Circle C	Antifriction bearings required to operate in a high vacuum.

The remaining materials are obvious choices for the intended application, i.e., OFHC copper for low temperature electrical conductors, tantalum for heating elements and shielding, and aluminum bronze for sleeves.

During the quarter, quotations for the drive motor and the synchronous permanent magnetic clutch were received. A special, thymatrol drive (5 hp output) with manual speed control, accurate to 2% of maximum speed, meets the requirements of the tester most adequately. Based on studies performed at General Electric's Magnetic Materials Section, it is apparently feasible to transmit 5 hp at maximum design speed by modifying the inside and outside diameters of their commercially available twenty-tooth permanent magnets.

The high vacuum chamber manufactured for and purchased by the General Electric Company for the program has been checked out successfully at a 6 x 10^{-10} torr vacuum using a General Electric Bayard-Alpert ionization gauge. To achieve this pressure, the pump-down time was within 15 hours. As an indication of the low leak rate of the system in conjunction with a 18-inch diameter x 30-inch high chamber, five days after the power to the pump was shutoff, the pressure had increased from the 10^{-9} torr to the 10^{-8} torr range. Within several hours after the power was restored, the pressure again dropped to the 10^{-9} torr range and subsequently to 3 to 4 x 10^{-10} torr.

Friction and Wear in Liquid Potassium

Design of the liquid potassium version of the friction and wear tester was initiated and currently is in the final stages. The design that has been developed should meet the critical requirements of this phase of the program. One design criterion was that the test specimen configuration and contact area of both the liquid and dry version of the testers be identical. From the standpoint of obtaining comparable data with potassium as a lubricant and without a lubricant in a high vaccum, this is important.

VII. FUTURE PLANS

The summary procedure which follows enumerates the steps to be pursued during the succeeding quarter to implement this study.

1) Continue the literature search and the compilation of property data on candidate materials.

2) Receive the seamless Cb-1Zr tubing for the capsule corrosion test program.

3) Complete procurement specifications and order test specimens of candidate materials.

4) Initiate construction of the corrosion and the dimensional stability test facilities.

5) Conduct checkout tests on the hot hardness and thermal expansion apparatus.

6) Complete construction of the compression test facility.

7) Complete detail design of the liquid potassium friction and wear test rig and initiate detailed manufacturing drawings.

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